

Friday Morning, October 6, 2000

Manufacturing Science and Technology Room 304 - Session MS-FrM

Langmuir Award/Ultra Clean Society and Contamination Free Manufacturing

Moderator: A. Diebold, Sematech

8:20am **MS-FrM1 Surfaces, Interfaces, and Chemical Reactions in Semiconductor Technology and Manufacturing, G.W. Rubloff**, University of Maryland **INVITED**

Atomic-scale properties and chemical reactivity of surfaces and interfaces constitute the science upon which semiconductor technology advances. Since thin film growth, etching, and modification occur as dynamic sequences of surface or interfacial reaction steps, surface reaction phenomena indeed determine the morphology, topography, microstructure, and chemical/physical properties of microelectronic structures, as well as their resulting functionality in devices, circuits, and systems. The surface science community brings an invaluable set of research strategies to understand, control, and advance semiconductor technology, particularly in combining vacuum technology and highly controlled process conditions together with surface, interface, gas phase, and thin film materials characterization. These synergies are proving effective as well in addressing key issues in semiconductor manufacturing.

9:00am **MS-FrM3 The Application of In-situ Monitor of Extremely Rarefied Particle-clouds Grown Thermally Above Wafers by using Laser Light Scattering Method to the Development of the Mass-production Conditions of the Tungsten Thermal CVD, N. Ito, T. Moriya, F. Uesugi**, NEC Corporation, Japan; *S. Mariya, M. Aomori, Y. Kato*, Tokyo Electron Ltd., Japan; *M. Tachibana*, Tokyo Electron Yamanashi Ltd., Japan

It is successfully demonstrated that the scattered-light intensity by thermally grown particle-clouds above wafers in the real tungsten (W) CVD chamber has a good correlation both with the surface roughness of the W-CVD film measured by the atomic force microscopy (AFM) and with the raw material gas-flow ratio SiH_4/WF_6 . In addition, since our in-situ particle monitor above wafers can detect particle-clouds consisting of particles less than 10nm in size, the appearance and the motion of particle-clouds corresponding to the transient variation of the ratio SiH_4/WF_6 at the conversion of gases and/or at the change of the flow rate can be observed. On the basis of these results, the mass-production conditions of particle-free and smooth surface of W-films are clarified with short Cycle Time. The traditional ways of developing the mass-production CVD conditions need many monitor-wafers and are time consuming, because both clarification of the suppression conditions of gas-phase particles by the in-situ particle monitor in vacuum exhaust lines and confirmation by the wafer-level visual inspection are indispensable. Moreover, the sensitivities of in-situ particle monitor at an exhaust line and the wafer-level inspection machine become insufficient to develop process conditions, as reduction of design rule for LSI proceeds. Therefore, application of the in-situ particle monitor above wafers to the development of mass-production conditions is notable method to minimize the non-product wafers and to realize short Cycle Time.

9:20am **MS-FrM4 Standard Practice for Temperature Calibration of the Silicon Substrate in Temperature Programmed Desorption Analysis, T. Matsunaga**, Matsushita Inc., Japan; *N. Yabumoto*, NTT Adv. Tech. Co., Japan; *N. Hirashita*, Oki Electric Ind. Co., Ltd., Japan; *H. Okumura*, Toray Res. Center, Inc., Japan; *M. Nishiduka*, Toshiba Corp., Japan; *I. Nishiyama*, NEC Corp., Japan; *M. Matsuura*, Mitsubishi Corp., Japan; *M. Morita*, Osaka Univ., Japan; *A. Shimazaki*, Toshiba Corp.; *T. Jimbo*, Hitachi, Ltd.; *T. Ajioka*, NTT Electronics Corp.

Ultra Clean Society (UCS)'s Equipment Standardization Working Group proposed a standard practice covering temperature calibration of the silicon substrate, ranging from 400 to 1000°C, for temperature programmed desorption (TPD) analysis. Although TPD has been widely used to characterize materials and fabrication processes in ULSI devices, the temperature is not accurate enough to develop reliable fabrication processes. The desorption temperature was found to differ over 100°C between interlaboratory TPD measurements. In order to solve this problem, the ramping temperature of TPD instrument was calibrated to silicon substrate temperature by this proposed standard practice, which consists of heating silicon calibration materials at a controlled rate in TPD

instrument, measuring characteristic desorption peak temperatures, and quadratic calibration fitting to the standard temperatures. The calibration materials are (1) CaCO_3 dropped on a Si wafer and dried, (2) Ar and (3) H ion implanted into Si wafers. The standard temperatures of the characteristic desorption, associated with decomposition, structural transformation and lamination of silicon, were determined by special TPD instrument with the highest isothermal space around the specimen among the interlaboratory. The precision of this practice was determined in an interlaboratory test in which 4-5 laboratories participated using two instrumental models. This test using a few common specimens proved that the average standard deviation, measured in different laboratories for all the measurements with ramping rates of 10, 30 and 60°C/min, was estimated to be 6.0°C between 400 and 1000°C.

9:40am **MS-FrM5 Standardization of the Method to a Disiloxane Concentration in Monosilane Gas using Atmospheric Pressure Ionization Mass Spectrometer, M. Kitano**, Tohoku Univ., Japan; *Y. Sakakibara*, NTT Adv. Tech. Corp., Japan; *Y. Ishihara*, Nippon Sanso Corp., Japan; *Y. Kunii*, Kokusai Electric Co., Ltd., Japan; *K. Hasumi*, Hitachi Tokyo Electronics Co., Ltd., Japan; *I. Matsuda*, Syowa Denko K.K., Japan; *A. Ohki*, Osaka Sanso Kogyo Ltd., Japan; *Y. Shirai*, Tohoku Univ., Japan

Ultra clean society (UCS)'s process gas measurement standardization working group proposed, using atmospheric pressure ionization mass spectrometer (APIMS) which has two ionization compartments to measure the disiloxane ($\text{SiH}_3\text{-O-SiH}_3$) in monosilane (SiH_4) gas at concentrations between 10ppb to 1000ppb. In this standard, SiH_4 with $\text{SiH}_3\text{-O-SiH}_3$ of unknown concentration is introduced into the second ionization compartment. Ar^+ ion generated by corona discharge in the first ionization compartment is sent out to the second ionization compartment. And Ar^+ ion reacts with $\text{SiH}_3\text{-O-SiH}_3$ in the SiH_4 gas to exchange ion, and $\text{SiH}_3\text{-O-SiH}_3$ ion are generated in large amount. $\text{SiH}_3\text{-O-SiH}_3$ is detected with mass number of 77. Using relative ion intensity of $\text{SiH}_3\text{-O-SiH}_3$ and calibration curve acquired by follow method, $\text{SiH}_3\text{-O-SiH}_3$ concentration is determined. H_2O of a known concentration firstly adsorbed into the stainless steel tube. This tube is purged out using SiH_4 gas. Adsorbed H_2O reacts with SiH_4 , and $\text{SiH}_3\text{-O-SiH}_3$ is generated. Adsorbed H_2O volume and generated total disiloxane volume are in a proportion of one to one. So that the amount of disiloxane in SiH_4 gas can be quantitatively calculated. The determination limit, which was defined as 3 times of the standard deviation of $\text{SiH}_3\text{-O-SiH}_3$ concentration in purified SiH_4 gas was found to be 10ppb. For verification of calibration curve, calibration curves, which were prepared at different timings and different places by different people, showed good agreement of over 80%. Moreover, it is proved that calibration curve of $\text{SiH}_3\text{-O-SiH}_3$ in SiH_4 can be substituted by that of H_2O in Ar which is corrected with a correction coefficient.

10:00am **MS-FrM6 A Wide Range Vacuum Sensor Fabricated by MEMS, H. Miyashita, Y. Kitamura, H. Watanabe**, ANELVA Corporation, Japan; *M. Esashi*, Tohoku University, Japan

A wide range capacitive vacuum sensor has been developed by microelectromechanical system (MEMS) technology. Use of MEMS technology has many advantages such as ability to miniaturization of the sensor, mass production, cost reduction etc. when compared to the conventional mechanical processes. The reason is that MEMS process is very similar to that of semiconductor device fabrication processes. The sensor is comprised of an SD2 glass and a silicon substrate. The length, width and thickness of the sensor is 20mm, 20mm, and 1.4mm, respectively. The fabricated vacuum sensor has two silicon diaphragms with 4mm x 4mm and 1mm x 1mm dimensions, and the thickness of the diaphragms is 7µm. Since the deflection of a diaphragm depends on the diaphragm size, the sensor measures a wide range of pressure. The smaller diaphragm shows linear characteristics of electrostatic capacitance at the pressures below 40,000Pa. The larger diaphragm shows linear characteristics of electrostatic capacitance at the pressures below 200Pa. The developed sensor is able to measure pressure in the range of 0.04Pa to 40,000Pa. This is achieved by using an electrical circuit which converts electrostatic capacitance into voltage over three orders. The same technology is applicable to the fabrication of vacuum sensors that measure pressures in other ranges by changing the size of the diaphragms. Moreover, the number of diaphragms in a sensor also can be increased to

¹ Gaede-Langmuir Award Winner

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enhance the pressure-monitoring region without increasing the fabrication processing steps.

10:40am **MS-FrM8 The Effect of Molecular Weight of Organic Contaminants on their Adsorption on Si-wafers**, *Y. Wakayama*, Tohoku University, Japan; *S. Kobayashi*, *T. Ishii*, Taisei Corporation, Japan; *S. Sugawa*, *T. Ohmi*, Tohoku University, Japan

It is well known that organic contaminants adsorbed on silicon wafer surface degrade the performance of ULSI devices. However, there have been no reports on relation of adsorption behavior of organic contaminants to the silicon or silicon oxide surfaces and their molecular weight. In this study, we have found that the amount of adsorbates on a silicon oxide surface depends on the molecular weight of organic substances. In order to investigate the adsorption behavior of organic contaminants on silicon oxide surfaces, we used solid waxes with aliphatic hydrocarbons, polyvinyl chloride sheets with phthalic esters and silicone sealing materials containing cyclosiloxanes with different molecular weights (Mw) as a contamination source. Each of these materials was separately stored in a closed vessel with a wafer with oxide film for 24 hours. The above organic adsorbates on the oxide surface were analyzed by thermally desorbed and gas chromatography-mass spectroscopy. It was found from our data that as the molecular weight of the organic compounds on the oxide surface increases the amount of adsorbates from oxide surface increases as well and reaches a maximum. Thereafter the amount gradually decreases down to the detection limit of the instruments. It is thought that the phenomenon is related in some way to the balance between the heat of adsorption of organic substances and the vapor pressure. Because, The heat of adsorption that determines the adsorption ability of organic compounds on Si-wafer increases with increase of Mw. On the other hand, the vapor pressure of organic compounds that also determine their rates of adsorption on Si-wafer decreases with increase of Mw.

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